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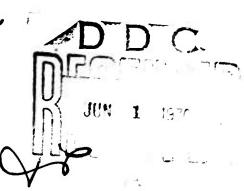
# MONSANTO/WASHINGTON UNIVERSITY ONR/ARPA ASSOCIATION

THE EFFECT OF TIME AND TEMPERATURE
ON THE MECHANICAL BEHAVIOR OF EPOXY COMPOSITES
PART II. MODE OF FAILURE, YIELD STRESS AND YIELD STRAIN

BY

A. E. MOEHLENPAH, O. ISHAI, A. T. DIBENEDETTO

PROGRAM MANAGER
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January 1970

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#### FOREWORD

The research reported herein was conducted by the staff of the Monsanto/Washington University Association under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 876, ONR contract authority NR 356-484/4-13-66, entitled "Development of High Performance Composites."

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#### **Abstract**

A crosslinked epoxy resin consisting of a 60/40 weight ratio of Epon 815 and Versamid 140 and composites of this material with glass beads, unidirectional glass fibers and air (foams) were tested in tension, compression and flexure to determine the effect of time and temperature on the mode of failure, yield stress, and yield strain. Unidirectional continuous fiber-filled samples were tested at different fiber orientation angles with respect to the stress axis. Strain rates ranged from 10<sup>-4</sup> to 10 min<sup>-1</sup> and the temperature from -1 to 107°C.

The material was found to change from a brittle-to-ductile-to-rubbery failure mode with the transition temperatures being a function of strain rate, filler content, filler type and fiber orientation angle, indicating that the transition is perhaps dependent on the state of stress.

In the ductile region, an approximately linear relationship between yield stress and log strain is evident in all cases. The isotherms of yield stress versus log strain rate

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were shifted to form a practically linear master plot of yield stress versus log shifted strain rate that can be used to predict the yield stress of the composites at any temperature and strain rate in the ductile region. The time-temperature shift factors were found to be independent of the type, concentration and orientation of filler and the mode of loading. Thus, the composite shift factors seem to be a property of the matrix and not dependent on the state of stress. The compressive-to-tensile yield stress ratio was practically invariant with shifted strain rate for the unfilled matrix, while fillers and voids raised this ratio and caused it to increase with a decrease in shifted strain rate. The yield strain of the composites is less than the unfilled matrix and is a function of fiber orientation and shifted strain rate.

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#### I. Introduction

The effect of time and temperature on the tangent modulus and stress relaxation of epoxy composites was discussed in Part I [1]. The mechanical behavior of the unfilled matrix was considered in another paper [2]. The effect of time and temperature on the mode of failure, yield stress, and yield strain of epoxy composites will be reported in this paper.

#### A. Failure Modes and Transitions

The failure modes were determined from the shape of the load versus deformation curves. The failure modes are schematically illustrated in Figure 1. A material is defined as "brittle" if it fails in the decreasing slope region before the stress-strain curve reaches a maximum. "Ductile" behavior is defined when the stress-strain curve exhibits a yield maximum. The term "ductile-rubbery" is applied when the stress-strain curve shows an inflection point but not a maximum. A mode is called "rubbery" if the stress-strain curves have constantly increasing slope but have no visible inflection or maximum.

#### B. Yield Stress

Nicholas and Freudenthal [3] showed that addition of NaCl filler to polyurethanes

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changes the stress-strain behavior from rubbery to ductile and that yield stress increases as the particle size decreases. Lohr [4] compared the time-temperature dependence of yield stress versus log strain rate with stress relaxation for polymethylmethacrylate, polyethylene terephthalate, polystyrene and polyvinyl chloride. Essentially all of the yield stress data could be superposed to give an almost straight line which is described by the equation:

$$\sigma_{y} = K_{1} + K_{2} \log (\dot{\epsilon} A_{T})$$
 (1)

where  $K_1$  and  $K_2$  are constants,  $\sigma_y$  = yield stress, and  $\dot{\epsilon}$  = strain rate. He found that . in general the yield stress shift factors were less than the stress relaxation shift factors. His stress relaxation experiments were determined at 1.43 percent strain, whereas yield stress experiments were at higher strains where non-linearity and crack propagation probably occurred. This hinted that his  $A_T$  may perhaps be a function of stress or strain as well as temperature or that a different mechanism for flow occurs at different levels of stress or strain.

Ishai [5] carried out a series of loading tests on 1:1 weight ratio Epon 815–

Versamid 140 samples at a series of constant strain rates under tension, compression, and flexure at room temperature. In all cases the yield stress versus log strain rate was linear. The ratio of compressive-to-tensile yield stress ranged from 1.27 to 1.38. A series of creep tests was also performed on this material at high stress levels under tension, compression, and flexure [6]. In all cases there is linearity between creep stress and log secondary creep rate, which is almost coincident with the corresponding relationship between yield stress and log strain rate obtained in the constant strain rate tests. The deformation during yielding which was not recoverable at room temperature was almost completely recoverable when placed in an oven at temperatures above 100°C.

Ishai and Cohen [7] studied the effect of sand filler and air voids on the compressive yield of epoxy composites at room temperature. For all volume fractions (to 51 percent for sand and to 66 percent for voids) plots of yield stress versus log strain rate were linear. The slopes of these plots were the same for all concentrations of filler but decreased with increasing volume fraction of voids.

The effects of temperature and strain rate on the failure characteristics of transverse and longitudinal unidirectional glass-reinforced Epon 815-Versamid 140 were studied by Preis [8]. Part of these data has been included in this paper.

#### II. Experimental Procedure

The constituent materials, fabrication of test specimens, and testing procedure were described in Part 1 [1].

#### III. Results

#### A. Transition Behavior

The stress-strain behavior for the unfilled 60/40 Epon 815-Versamid 140 and composites fabricated using this matrix is dependent on temperature and strain rate. The transition regions for the unfilled and particulate filled material in terms of temperature and strain rate for compression are shown in Figure 2. The transition from ductile to rubbery behavior is about the same for both compression and tension. It was difficult to determine this transition in flexure since at high temperatures the deflection is so large that the sample begins to slip off the holder and the resulting load-deflection curves are invalid. Particulate filler or voids narrow the ductile region compared to

the unfilled matrix. The effect of particulate filler, voids, and continuous transverse fibers on the tensile brittle-ductile transition is shown in Figure 3. Flexural brittle-ductile transitions are practically coincident with tensile brittle-ductile transitions.

Fillers and voids cause the material to be brittle at higher temperatures and lower strain rates than the unfilled matrix. Figure 4 indicates that particulate filler or voids do not change the compressive ductile to ductile-rubbery transition much but that continuous transverse fibers raise this transition significantly. In summary, (1) the general patterns of transition behavior are very similar for all systems in these modes of loading, with the transition zones being a function of temperature and strain rate; (2) in general, the addition of non-longitudinal reinforcement will make the region of ductile behavior narrower; and (3) the brittle-ductile transition is much more sensitive to the presence of a reinforcement than is the ductile to ductile—upbery transition.

#### B. Yield Stress

In this paper the yield point is defined as the maximum in the load-deflection curve. Yield stress for all of the materials in all three modes of loading is dependent on temperature and strain rate. Figures 5 to 8 show, respectively, the effect of temperature and strain rate on the yield stress in tension and compression for the particulate filled, tension for continuous filled at 20° orientation, and compression for continuous transverse. These drawings are typical of others that could have been shown for other modes of loading, other angles of orientation, or the other systems.

For all modes of loading and for all types of materials tested, yield stress is practically linear with the logarithm of strain rate at constant temperature. Figure 9

shows compressive yield stress at a strain rate (0.2 min<sup>-1</sup>) for unfilled, particulate filled, continuous transverse filled, and foam\*. In general, the yield stress decreases with an increase in temperature. The effect of temperature on yield stress for other strain rates and for other modes of loading was very similar to that shown in Figure 9.

The yield stress versus log strain rate data was shifted along the strain rate axis to obtain yield stress master curves in compression, tension, and flexure. Fifty degrees Centigrade was chosen as a reference temperature because at this temperature most of the samples tested were ductile at the strain rates utilized. Values of the yield stress at 50°C were obtained from plots of yield stress versus temperature such as Figure 9. Figures 10 to 12 are plots of the yield stress shift factors versus temperature for the unfilled, particulate filled, continuous transverse filled, and foam samples in the three modes of loading. Clearly, neither the type of filler nor the mode of loading significantly affects

$$\sigma_{\text{pormalized}} = \sigma_{\text{yactual}} \left[ \frac{1 - 1.2 \times .241^{2/3}}{1 - 1.2 \, \text{C}_{\text{V}}^{2/3}} \right] \tag{2}$$

The volume fraction of filler or voids was determined from the densities by the following equation:

$$C_{V} = (\rho_{c} - \rho_{m})(\rho_{f} - \rho_{m}) \qquad (3)$$

where  $\rho_c$  = density of composite,  $\rho_f$  = density of filler, and  $\rho_m$  = density of matrix which was found to be 1.09 g/cc at room temperature.

<sup>\*</sup>Since the density of the foam from one sample to the next was not the same, the values of compressive yield stress were normalized to the average void fraction of 0.241 by the following equation derived from the work of Ishai [7]:

the yield stress shift factors. The yield stress shift factors from Figures 10 to 12 were curve-fit by a least-squares-type method to obtain the following cubic equation for log A<sub>T</sub>:

$$\log A_{Ty} = -.3149 - .1376 (T-50) - 4.952 \times 10^{-5} (T-50)^{2} + 7.196 \times 10^{-6} (T-50)^{3}$$
(4)

The root mean square deviation of log A<sub>T</sub> was 0.329. This equation was used to determine yield stress shift factors in subsequent figures. The tensile and flexural yield stress shift factors as a function of temperature for continuous filled samples at various fiber orientations are shown in Figures 13 and 14. In general, the shift factors are also independent of angle of orientation. The deviations that occur in flexural tests with the 20° orientation at low temperatures are probably due to shear coupling [9, 10]. The deviations appear when the length-to-width ratio of the flexure specimens was about 4. When tensile specimens with a length-to-width ratio of 12 were used, the shear coupling was minimized and the values of A<sub>T</sub> were reduced to the expected values.

A visual summary of the data is shown by the master curves of Figures 15 to 19. It can be seen in Figure 15 that the ultimate strengths reported in the brittle regions have a greater degree of scatter than the yield points in the ductile region and that the ultimate stress in the brittle zone is lower than the extrapolation of the yield stress line, indicating that failure occurred before the yield point was reached. Composites using 0.56 ± 0.01 volume fraction transverse continuous glass fibers have greater tensile, compressive and flexural yield stress than the unfilled matrix. Particulate glass filler increases the compressive yield stress but, at least at the 24 percent volume fraction, does not significantly improve the tensile and flexural yield stress. The presence of

voids lowers the tensile, compressive and flexural yield stress except at high temperatures in compression. The yield stress versus log shifted strain rate is practically linear over the entire ductile range for each composite and each mode of loading. However, a second-order equation of the form,

$$\sigma_y = K_1 + K_2 \ln (\epsilon A_T) + K_4 (\ln \epsilon A_T)^2$$
 (5)

was found to fit the data better than a linear equation. The Eyring [11, 12] theory of non-Newtonian viscous flow\* is a reasonable first approximation to the experimental

$$V_0 = \frac{4k\tilde{l}}{\log e K_2}$$
 (6)

where k = Boltzmann constant and K<sub>2</sub> = slope of yield stress versus log strain rate at T<sub>ref</sub>.

From plots of yield stress versus temperature at constant strain rate, one can determine the apparent activation energy, Q, and the frequency factor parameter, A, assuming that log (AT) is fairly independent of temperature.

$$\log (AT_{ref}) = \log \epsilon - \frac{K_3 T_{ref}}{K_2}$$
 (7)

where K<sub>3</sub> = slope at T of isochrone of yield stress versus temperature.

$$Q = \frac{V_0 \tilde{N}}{4} [K_1 + K_2 \log (AT_{ref})]$$
 (8)

where  $K_1 = yield stress at <math>T_{ref}$  at  $\dot{\epsilon} = 1$ , and  $\tilde{N} = Avagadro's number.$ 

It can be seen that the  $K_1$  and  $K_2$  are the same as that of Equation (1). The shift factor can be related [13] to the above parameters in the following way:

$$\log A_{T} = \log (AT_{ref}) (1 - \frac{T_{ref}}{T})$$
 (9)

<sup>\*</sup>The activation volume,  $V_0$ , can be determined at the reference temperature,  $T_{ref}$ , from the slope of the yield stress versus log strain rate plot at  $T_{ref}$ .

data, so using the first two terms of Equation (5) and isochrones of yield stress versus temperature, activation volumes, activation energies, and frequency factors were evaluated using Equations (6-8). Table I gives the results of these calculations. It can be seen that particulate filler and transversely oriented fibers do not affect the activation volumes much but the foam increases it considerably. This also can be seen from the slopes of Figures 15 to 17 since slope is inversely proportional to activation volume. The activation volumes determined in tension are about 1.3 times as large as those determined in compression. It can be seen that the term  $\log (AT_{ref})$  which is related to the shift factors remains relatively constant. Activation energies ranged from 63 to 89 kilocalories per mole.

The ratios of compressive yield stress to tensile yield stress are plotted as a function of shifted strain rate in Figure 20. The compressive to tensile yield ratio,  $\lambda$ , for the unfilled material remains fairly constant, ranging only from 1.12 to 1.28 over seven decades of shifted strain rate. For the particulate filled, continuous transverse filled and the foam,  $\lambda$  is greater than for the pure material.

With the appropriate values of  $\lambda$ , and the flexural yield stress, one can calculate the yield in tension and compression using the following equations [5]:

$$\sigma_{y_{\dagger}} = (\frac{\lambda + 1}{2\lambda}) \frac{Pl_o}{BH^2}$$
 (10)

and

$$\sigma_{y_c} = (\frac{\lambda + 1}{2}) \frac{Plo}{BH^2}$$
 (11)

where P = yield force, Io = length of flexural sample, B = width of flexural sample, and H = depth of flexural sample.

The calculated values of the yield in tension and compression are represented by the dashed lines in Figures 21 and 22. For continuous transverse specimens, the value of flexure expected from simple beam theory was obtained from tensile data and appropriate values of  $\lambda$ , using the following equation:

$$\sigma_{\text{f simple beam}} = \left(\frac{3\lambda}{\lambda+1}\right)\sigma_{y_{t}}$$
 (12)

The values obtained from this equation are represented by the dashed line in Figure 23 and compared with the two experimental points that were obtained in flexure. Thus Figures 21 to 23 show that the proposed analysis provides a suitable representation of the stress distribution at yield in flexure for these epoxy composites and that one can therefore predict tension and compression from flexure data and  $\lambda$  or vice-versa.

Figure 24 shows the effect of angle on the normalized tensile yield stress at 45°.

The dashed line is based on a maximum distortional energy concept\* for ductile systems.

$$\eta = \frac{\sigma_{y_c}^{(\theta)}}{\sigma_{y_{90^\circ}}} = \frac{\sigma_{y_c}^{(\theta)}}{\sigma_{y_{45^\circ}}} = \frac{1}{\sin\theta (1 + 2\cos^2\theta)}$$
(13)

where  $\sigma_{\mathbf{y_c}}(\theta)$  = composite uniaxial yield stress when fibers are oriented  $\theta$  degrees with respect to the stress axis.

<sup>\*</sup>A theory used to predict the tensile strength of predominantly ductile unidirectional fibrous composites is based on the maximum distortional energy concept. According to this theory, yielding will occur when the second invariant of the deviatoric stress tensor exceeds a constant. This criteria assumes that the isotropic stress tensor does not affect yielding. Equation (13) gives the following relationship for the normalized tensile yield stress,  $\eta$ , for the case of unidirectional composites:

Figure 24 shows that as  $\log (iA_T)$  increases (increase in strain rate and/or decrease in temperature), the data seems to approach the maximum distortional energy approximation.

The effect of angle on the normalized flexural yield stress is shown in Figure 25.

A shear coupling effect due to anisotropy [9, 10] causes the flexural specimens to twist while being tested. This extra energy required to twist the sample causes the measured flexural off-axis yield stress to be high. The shear coupling at 20° fiber orientation is greater than at 45°, thus the normalized yield stress at 20° is higher than the theory predicts. From the results shown in Figures 14, 19 and 25, one may conclude that off-axis testing of short flexural specimens is not satisfactory at the angles where a high degree of anisotropy occurs.

#### C. Yield Strain

Although there was considerable scatter (± 10%) in the yield strain data, several conclusions can be made: (1) Yield strain increases (from 4-5% up to 7-8% for unfilled material) as the rubbery region is approached at low strain rates and/or high temperature. (2) Flexural yield strains determined by the simple beam theory (6-8% at room temperature to 12-14% as rubbery behavior is approached) were higher than those obtained from tensile and compressive measurements. This is probably due to the inadequacy of the simple beam theory at high deflections. (3) Fillers and voids lower the yield strain.

#### IV. Conclusions

Yield stress data of epoxy composites can be correlated by the time-temperature superposition principle to obtain master plots of yield stress versus log of shifted strain

rate. The time-temperature shift factors are not affected by the mode of loading nor the filler content, type, or orientation even though the actual values of yield stress, yield strain, and transition temperatures for failure modes are affected by these variables. Thus, the composite shift factors are a property of the matrix and not dependent on the state of stress.

#### Acknowledgment

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TABLE I. EYRING THEORY PARAMETERS

Yield Test	Sample	Ps:	K <sub>2</sub> psi	Psi/o <sub>K</sub>	Activation Volume A <sup>o3</sup> /molecule	log (A <sub>2</sub> T <sub>ref</sub> )	Activation Energy Kcal/mole
Tension	Unfilled	5700	1156	-139.8	5160	37.8	63.2
Compression	Unfilled	7295	1502	-217	3980	46.0	75.4
Tension	Particulate	5814	1035	-132.2	5770	40.0	67.5
Compression	Particulate	8812	1323	-203	4510	48.9	82.2
Tension	Foam	2641	465	-80	12840	54.3	38.8
Compression	Foam	4334	575	-74.7	10380	41.2	72.7
Tension	Fibers (90°)	8910	1114	-148.4	5360	41.7	73.5
Compression	Fibers (90°)	13696	1457	-183	4100	39.8	72.9
Tension	Fibers (20°)	15052	1386	-204	4310	46.2	84.5
Tension	Fibers (45°)	7992	905	-125	6595	43.3	7.97
Tension	Fibers (60°)	7300	926	-150	6445	51.0	87.1

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#### LIST OF FIGURES AND TABLE

- Figure 1. Typical Stress-Strain Curves Representing the Different Modes of Behavior up to Yield and Failure.
- Figure 2. Transitions in Failure Modes for Particulate Filled.
- Figure 3. The Effect of Fillers and Voids on the Tensile Brittle-Ductile Transition.
- Figure 4. The Effect of Fillers and Voids on the Compressive Ductile-Ductile Rubbery Transition.
- Figure 5. Tensile Yield Stress Versus Log Strain Rate for Particulate Filled.
- Figure 6. Compressive Yield Stress Versus Log Strain Rate for Particulate Filled.
- Figure 7. Tensile Yield Stress Versus Log Strain Rate for Continuous Filled, 20° Fiber Orientation.
- Figure 8. Compressive Yield Stress Versus Log Strain Rate for Continuous Filled, Transverse.
- Figure 9. Compressive Yield Stress Versus Temperature at Strain Rate = .2 ± .01 (Min)<sup>-1</sup> for Unfilled, Particulate and Continuous Transverse Filled, and Foam.
- Figure 10. Log Tensile Yield Stress Shift Factor Versus Temperature for Unfilled, Particulate and Continuous Transverse Filled, and Foam, T<sub>ref</sub> = 50°C.
- Figure 11. Log Compressive Yield Stress Shift Factor Versus Temperature for Unfilled, Particulate and Continuous Transverse Filled, and Foam, T = 50°C.
- Figure 12. Log Flexural Yield Stress Shift Factor Versus Temperature for Unfilled,
  Particulate and Continuous Transverse Filled, and Foam, T = 50°C.
- Figure 13. Log Tensile Yield Stress Shift Factor Versus Temperature for 20, 45, 60 and 90° Continuous Filled, T<sub>ref</sub> = 50°C.
- Figure 14. Log Flexural Yield Stress Shift Factor Versus Temperature for 20, 45, 60 and 90° Continuous Filled, T = 50°C.
- Figure 15. Tensile Yield Stress Versus Log Shifted Strain Rate for Unfilled, Particulate and Continuous Transverse Filled, and Foam, T<sub>ref</sub> = 50°C.
- Figure 16. Compressive Yield Stress Versus Log Shifted Strain Rate for Unfilled,
  Particulate and Continuous Transverse Filled, and Foam, T = 50°C.

- Figure 17. Flexural Yield Stress Versus Shifted Strain Rate for Unfilled, Particulate Filled, and Foam, T = 50°C.
- Figure 18. Tensile Yield Stress Versus Log Shifted Strain Rate for 20, 45, and 60° Continuous Filled, T<sub>ref</sub> = 50°C.
- Figure 19. Flexural Yield Stress Versus Log Shifted Strain Rate for 20, 45, 60 and 90° Continuous Filled, T<sub>ref</sub> = 50°C.
- Figure 20. Compressive-to-Tensile Yield Ratio Versus Log Shifted Strain Rate for Unfilled, Particulate and Continuous Transverse Filled, and Foam, Tref = 50°C.
- Figure 21. Yield Stress Versus Log Shifted Strain Rate for Particulate Filled, T = 50°C.
- Figure 22. Yield Stress Versus Log Shifted Strain Rate for Foam, T<sub>ref</sub> = 50°C.
- Figure 23. Yield Stress Versus Log Shifted Strain Rate for Continuous Transverse,  $T_{ref} = 50^{\circ}\text{C}.$
- Figure 24. Normalized Tensile Yield Stress Versus Fiber Orientation Angle.
- Figure 25. Normalized Flexural Yield Stress Versus Fiber Orientation Angle.

Table I. Eyring Theory Parameters

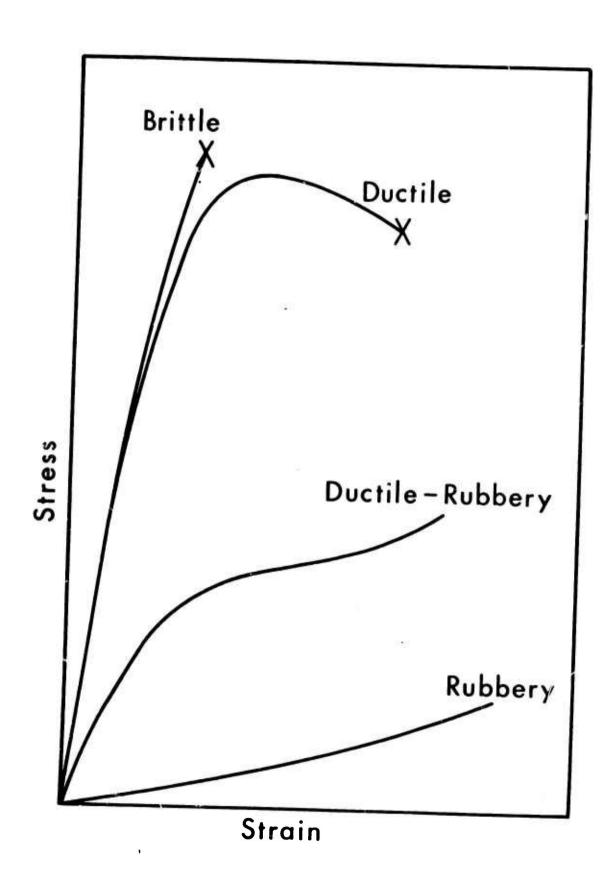


Figure 1 - Typical Stress-Strain Curves Representing the Different Modes of Behavior up to Yield and Failure

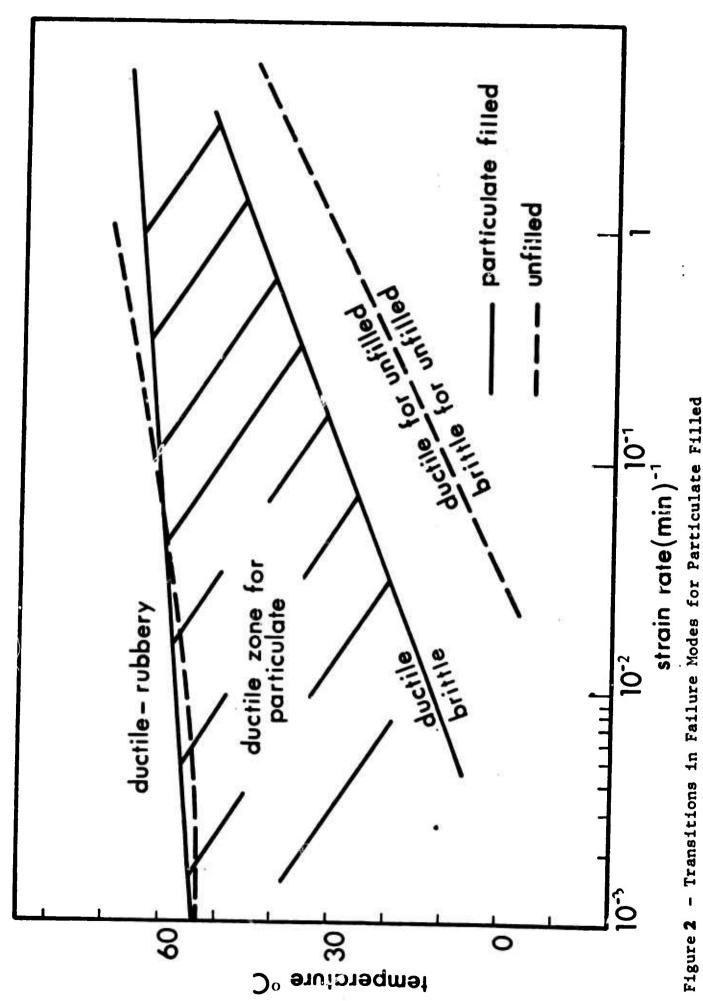


Figure 2 - Transitions in Failure Modes for Particulate Filled

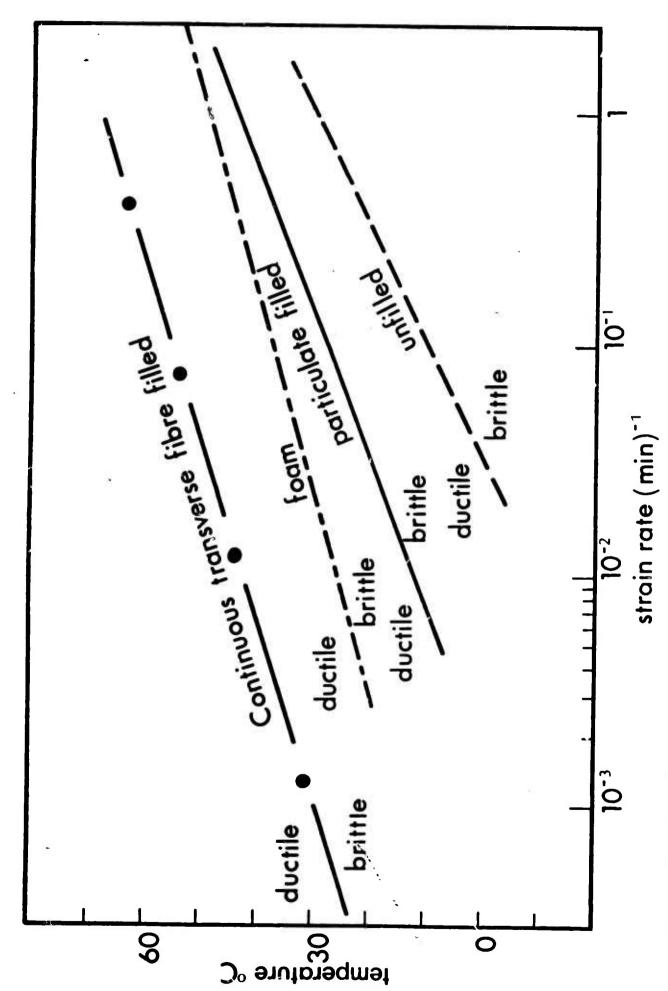


Figure 3 - The Effect of Fillers and Voids on the Tensile Brittle-Ductile Transition

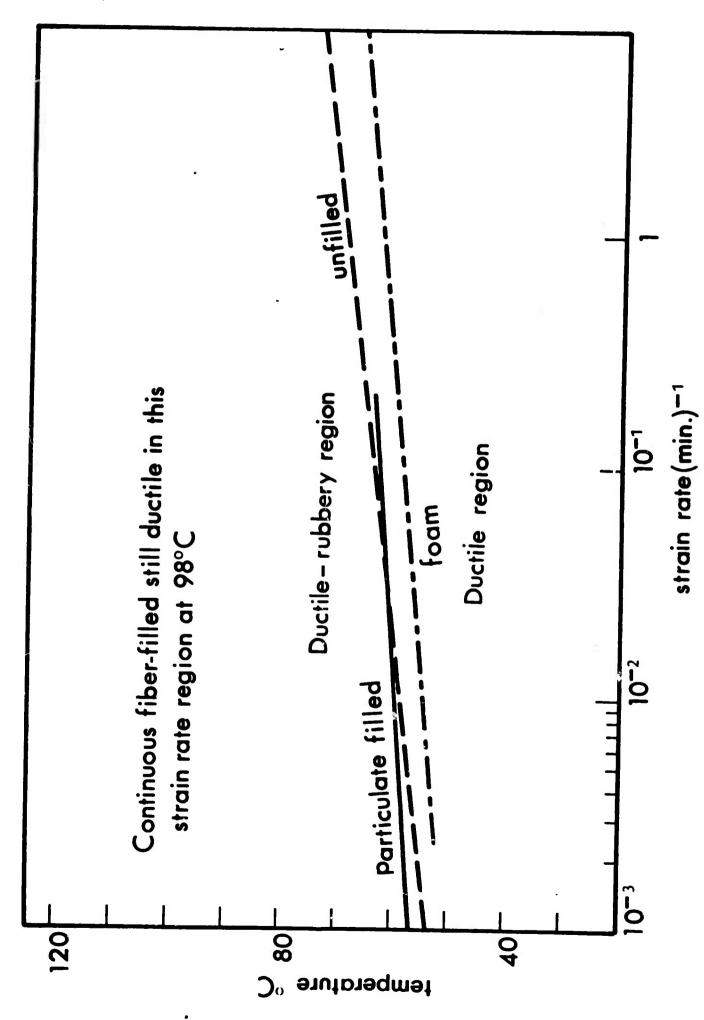
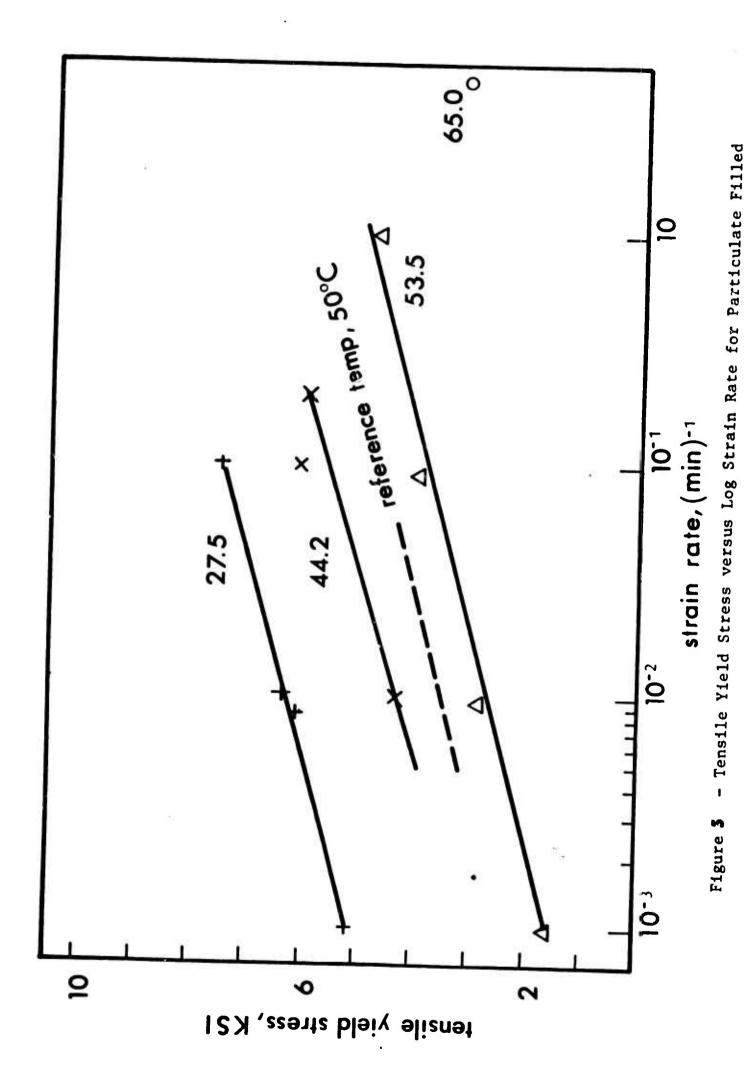
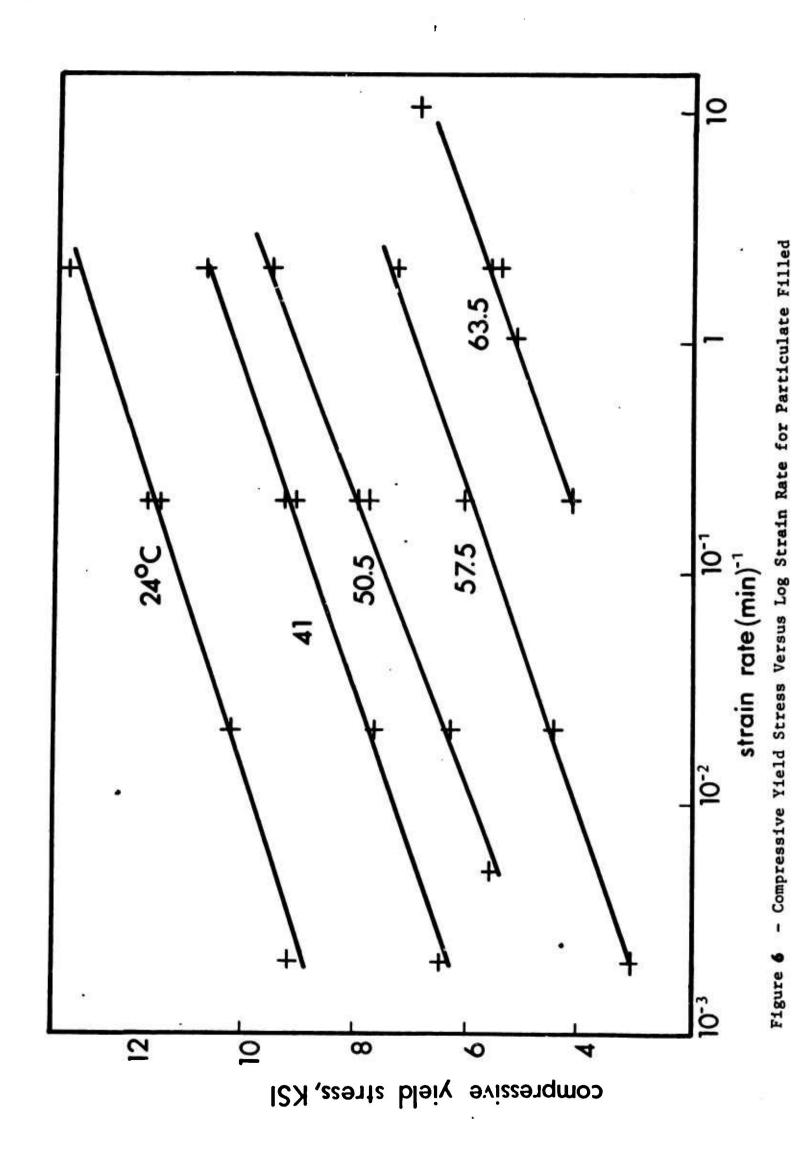
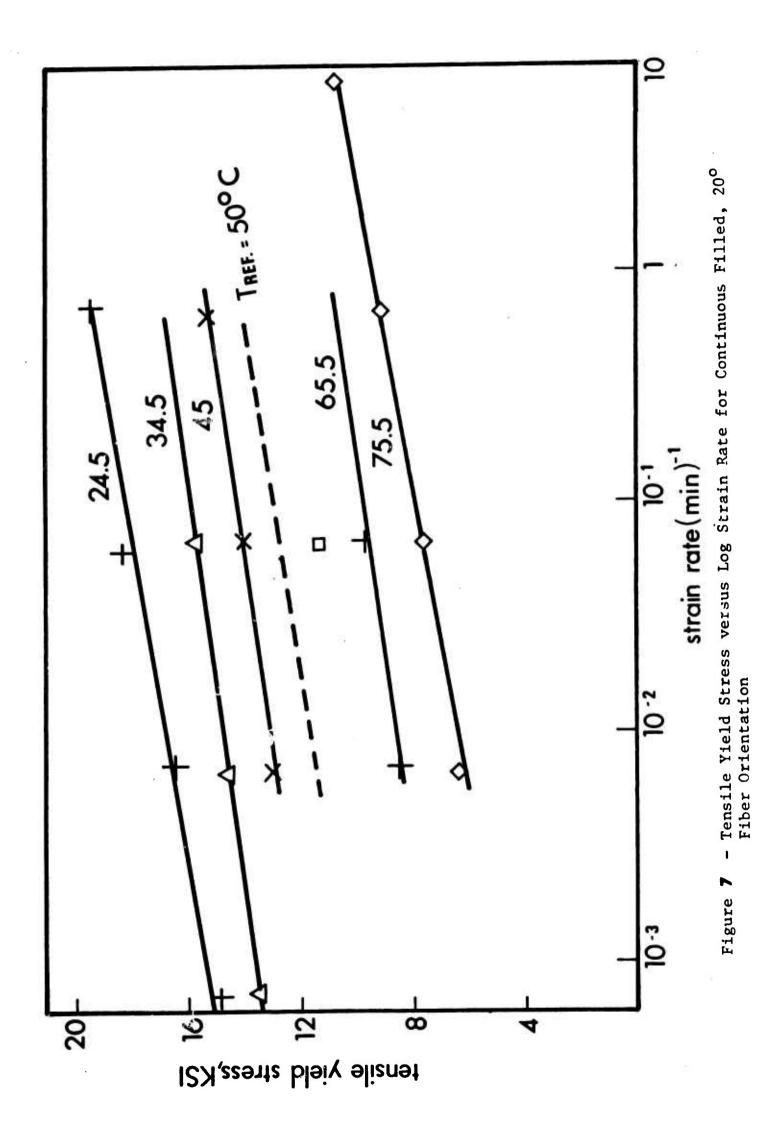
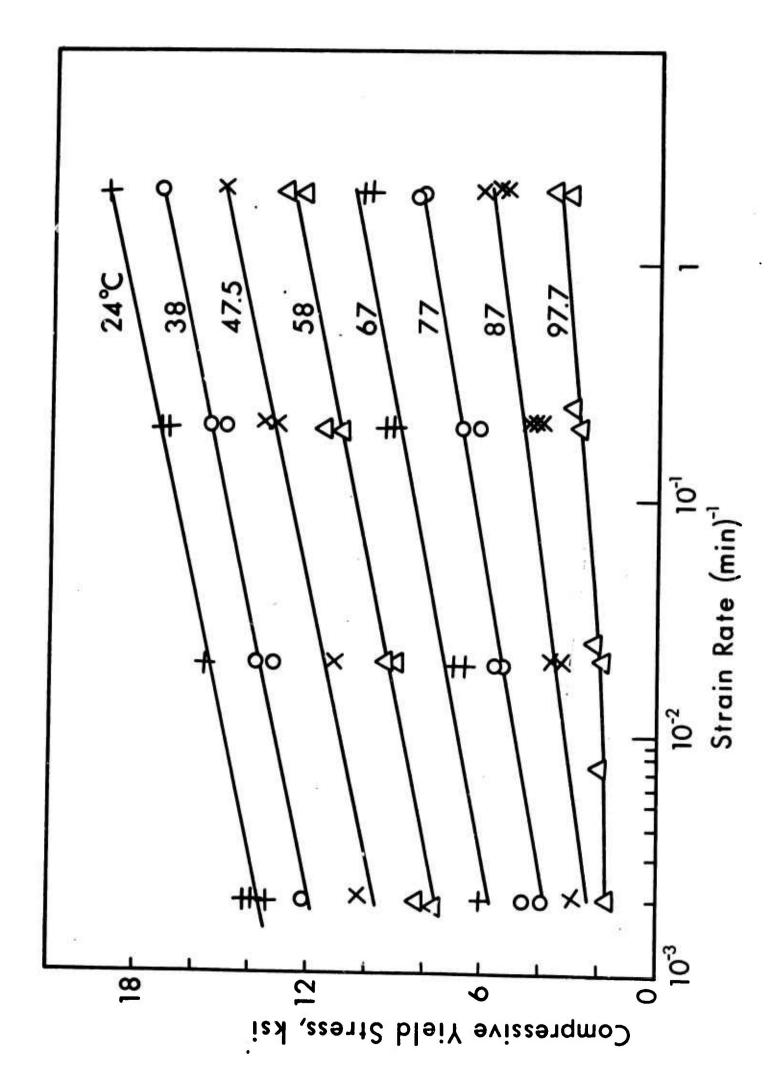


Figure 4 - The Effect of Fillers and Voids on the Compressive Ductile-Ductile Rubbery Transition









- Compressive Yield Stress versus Log Strain Rate for Continuous Filled, Transverse

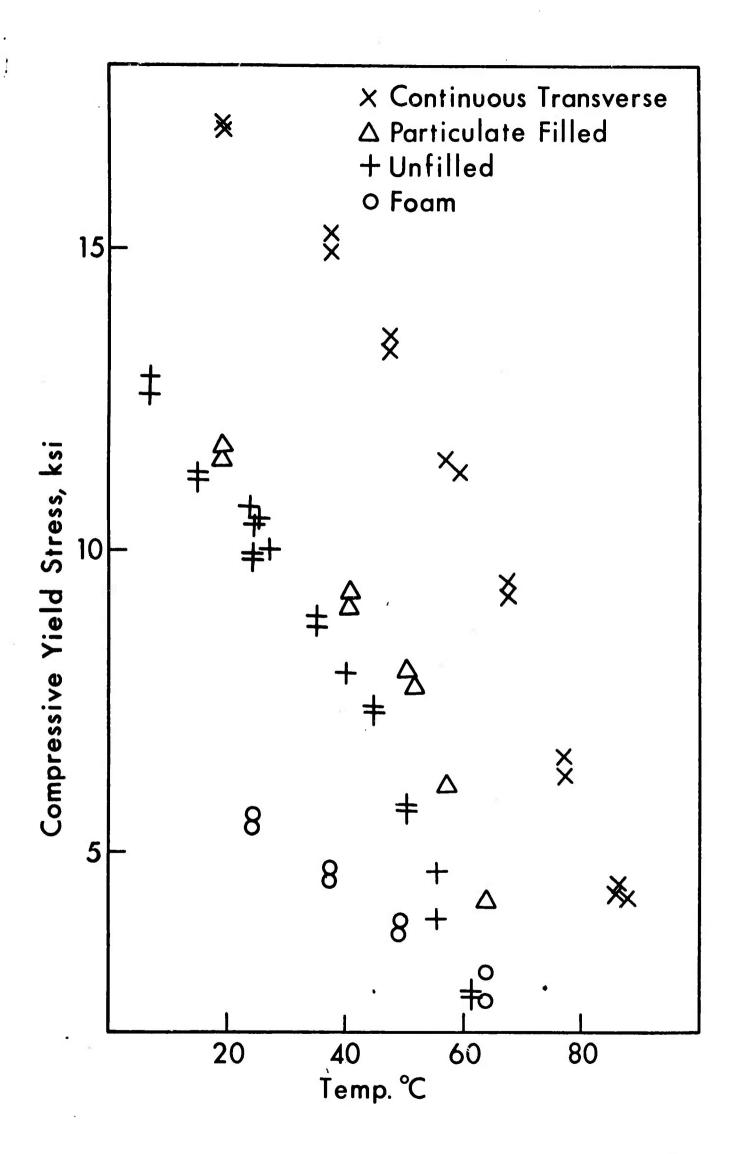


Figure 9 - Compressive Yield Stress versus Temperature at Strain Rate = .2 ± .01 (Min)<sup>-1</sup> for Unfilled, Particulate and Continuous Transverse Filled, and Foam

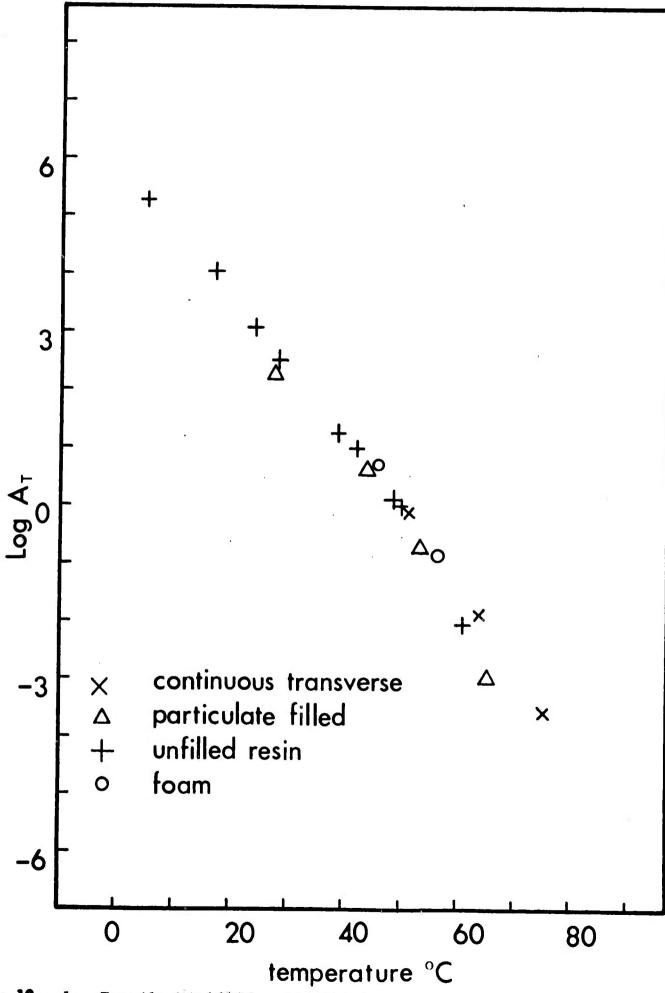


Figure 10 - Log Tensile Yield Stress Shift Factor versus Temperature for Unfilled, Particulate and Continuous Transverse Filled and Foam, Tref = 50 oc

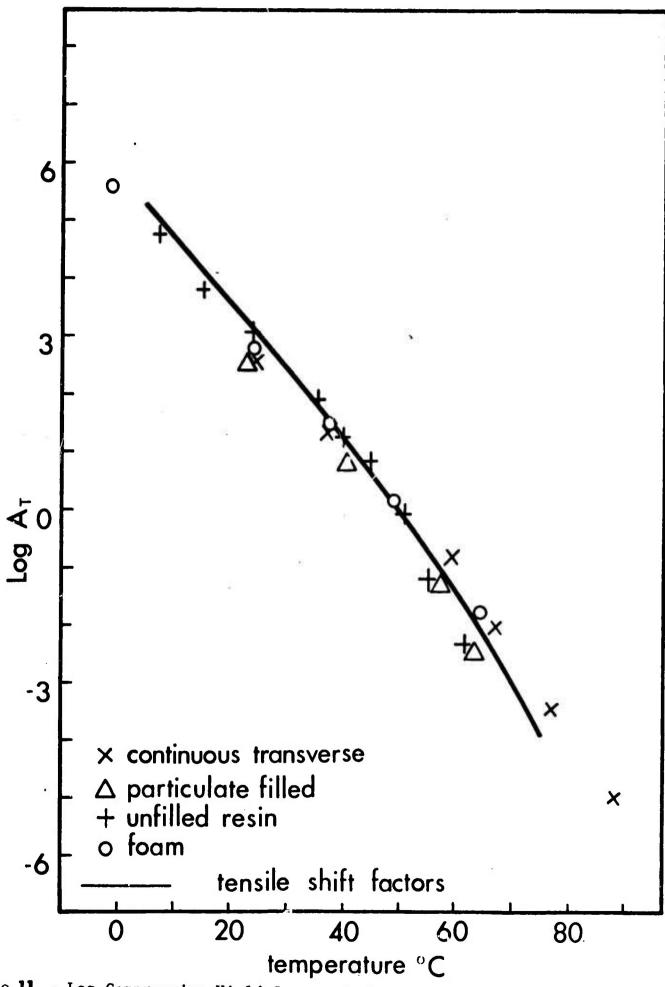


Figure 11 - Log Compressive Yield Stress Shift Factor versus Temperature for Unfilled, Particulate and Continuous Transverse Filled, and Foam,  $T_{ref} = 50 \, ^{\circ}\text{C}$ 

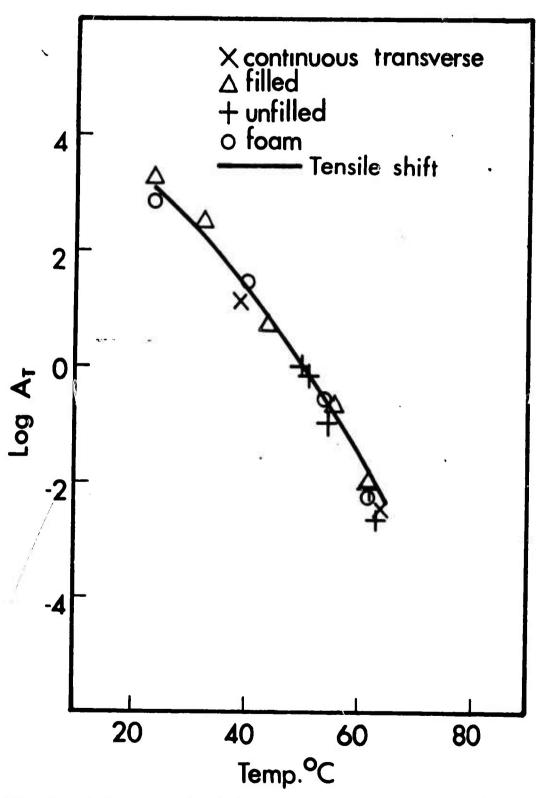


Figure 12 - Log Flexural Yield Stress Shift Factor versus Temperature for Unfilled, Particulate and Continuous Transverse Filled, and Foam,  $T_{ref} = 50$  °C

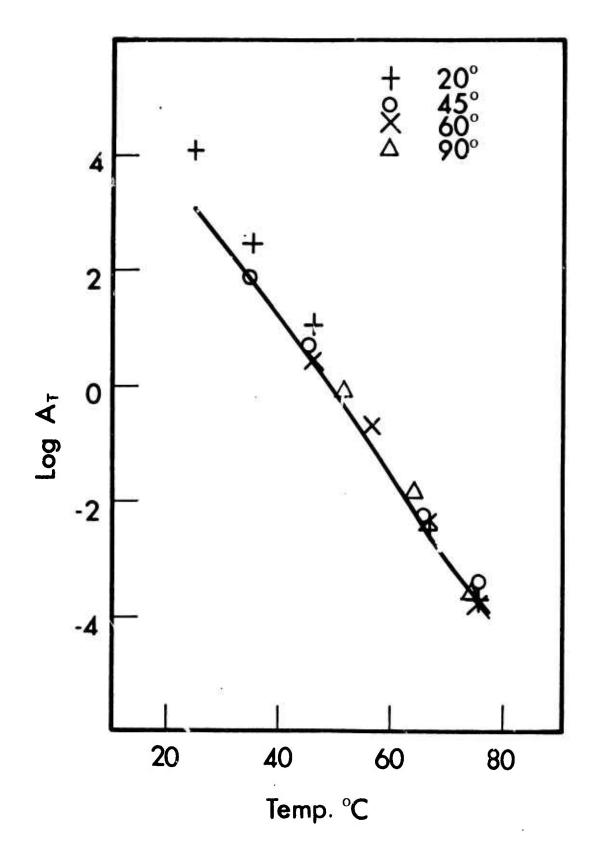
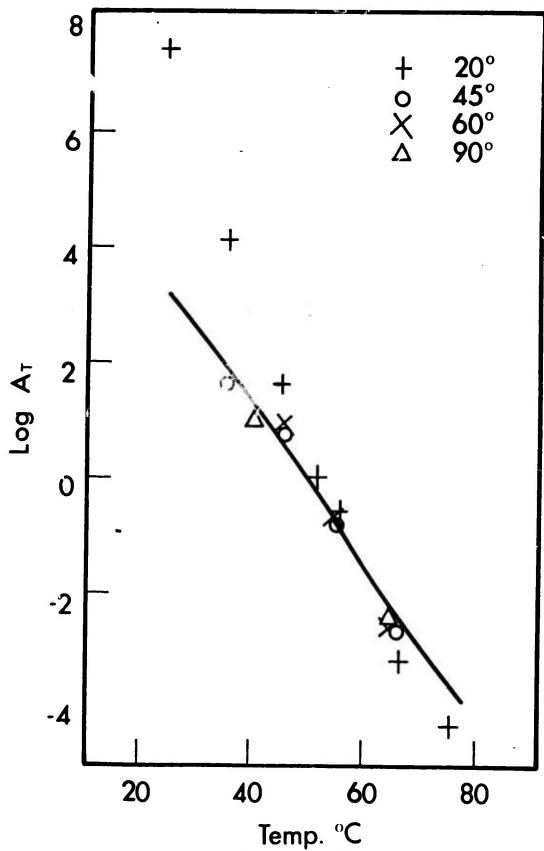


Figure 13 - Log Tensile Yield Stress Shift Factors versus Temperature for 20, 45, 60 and  $90^{\circ}$  Continuous Filled,  $T_{ref} = 50^{\circ}C$ 



Temp. °C

Figure 14 - Log Flexu: 11 Yield Stress Shift Factors versus Temperature for 20, 45, 60 and 90° Continuous Filled, T<sub>ref</sub> = 50 °C

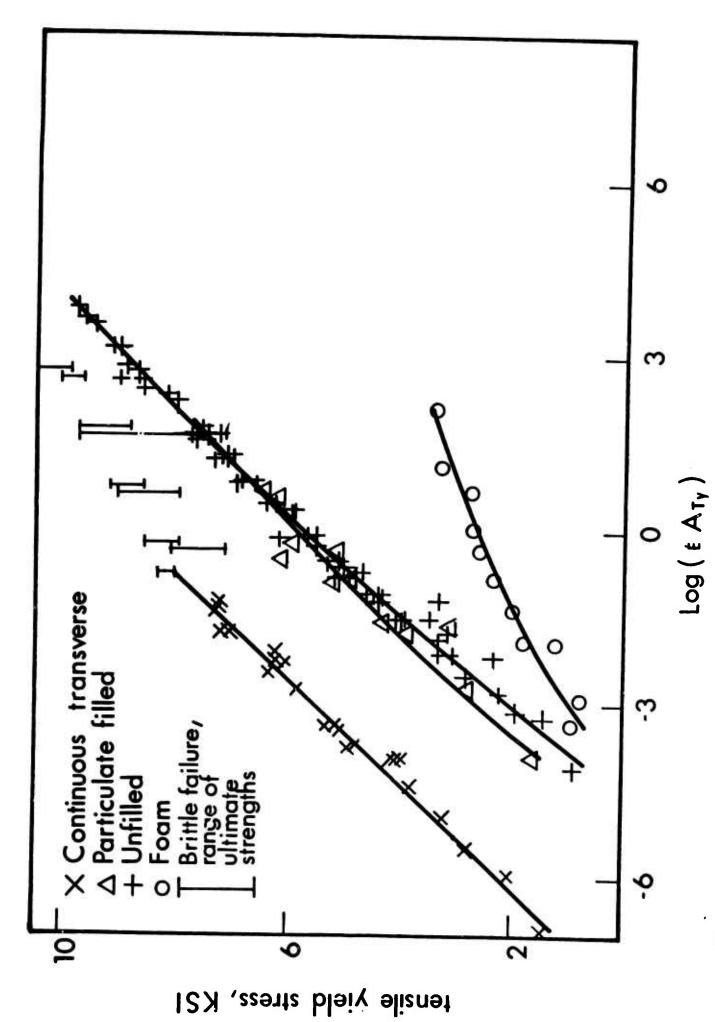


Figure 15 - Tensile Yield Stress versus Log Shifted Strain Rate for Unfilled, Particulate and Continuous Transvers? Filled, and Foam,  ${\rm Tref}$  = 50  $^{\rm O}{\rm C}$ 

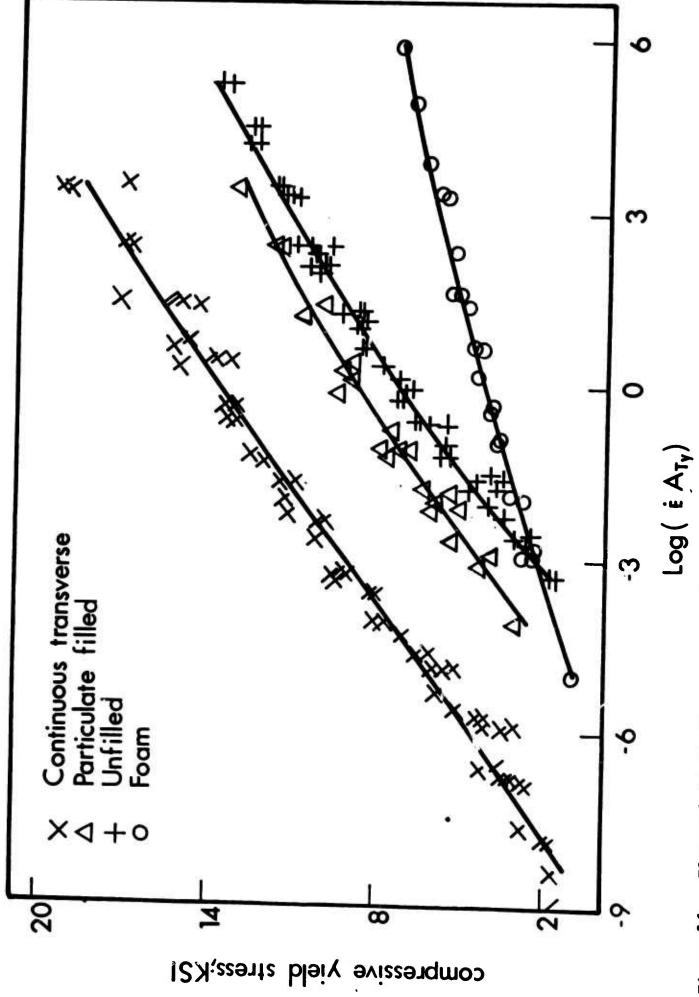


Figure 16 - Flexural Yield Stress versus Shifted Strain Rate for Unfilled, Particulate Filled, and Continuous Transverse Filled, and Foam,  $r_{\rm ref}$  = 50  $^{\circ}$ C

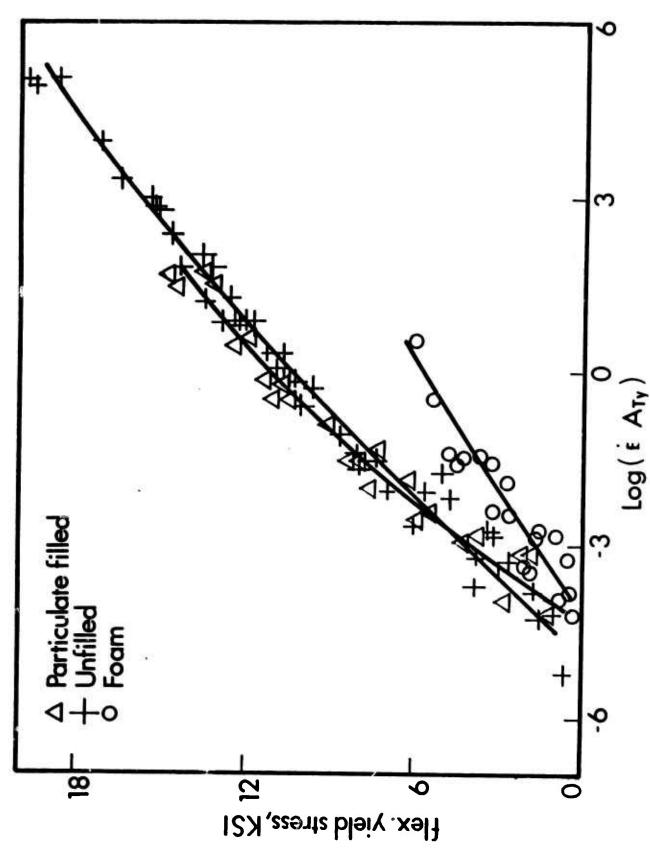
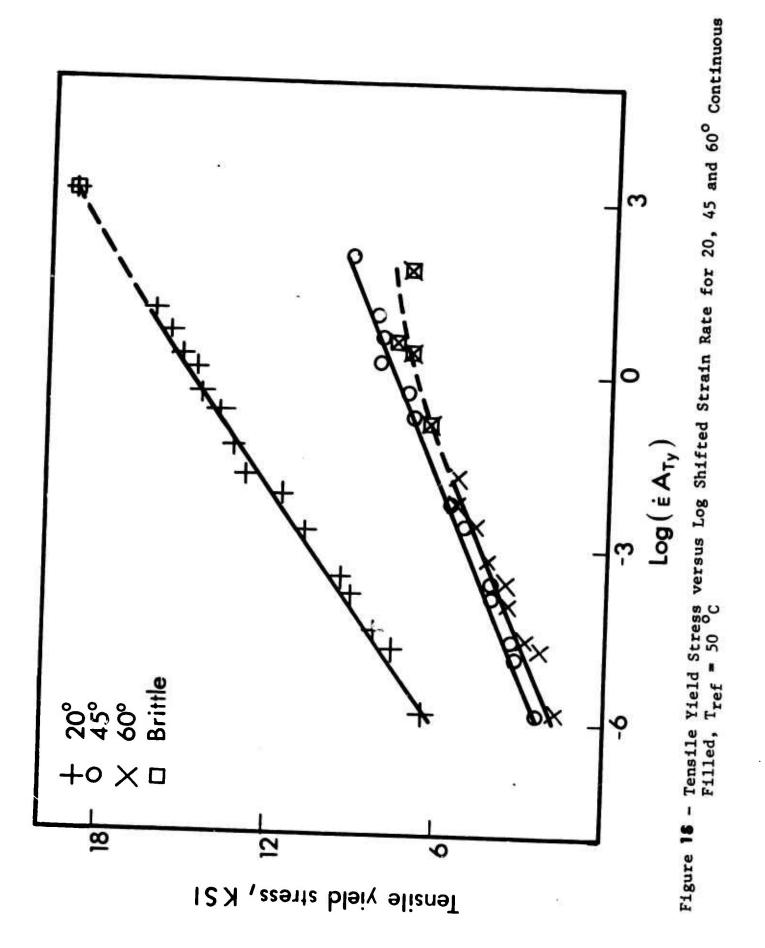


Figure 17 - Flexural Yield Stress versus Shifted Strain Rate for Unfilled, Particulate Filled, and Foam,  ${
m T_{ref}}$  = 50  $^{
m O}{
m C}$ 



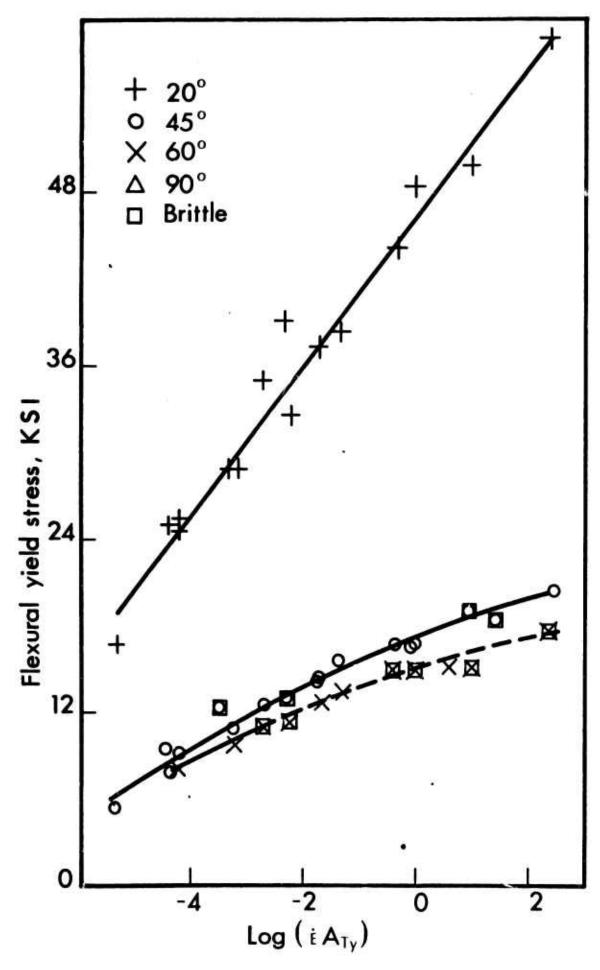


Figure 19 - Flexural Yield Stress versus Log Shifted Strain Rate for 20, 45, 60 and  $90^{\circ}$  Continuous Filled,  $T_{ref}$  =  $50^{\circ}$ C

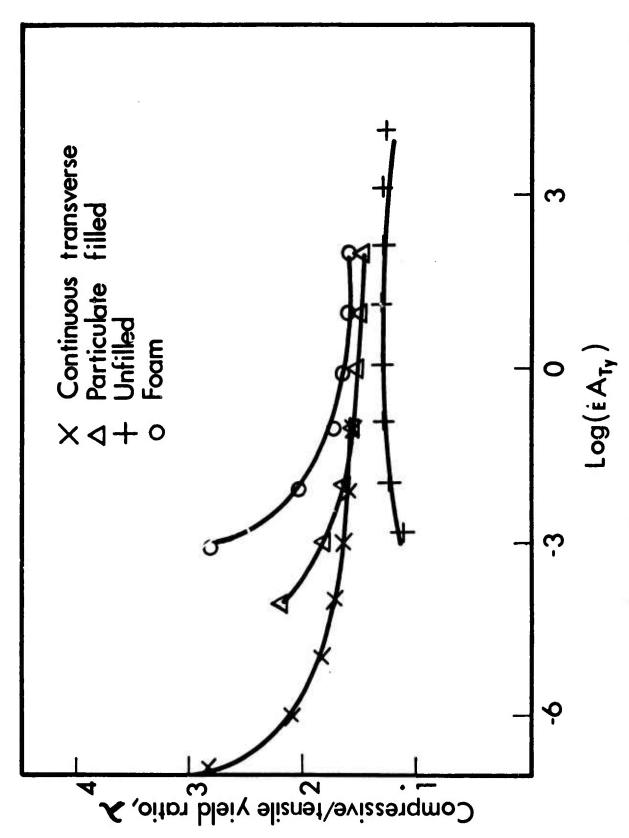


Figure 20 - Compressive to Tensile Yield Ratio versus Log Shifted Strain Rate for Unfilled, Particulate and Continuous Transverse Filled and Foam,  $T_{\rm ref}$  = 50 °C

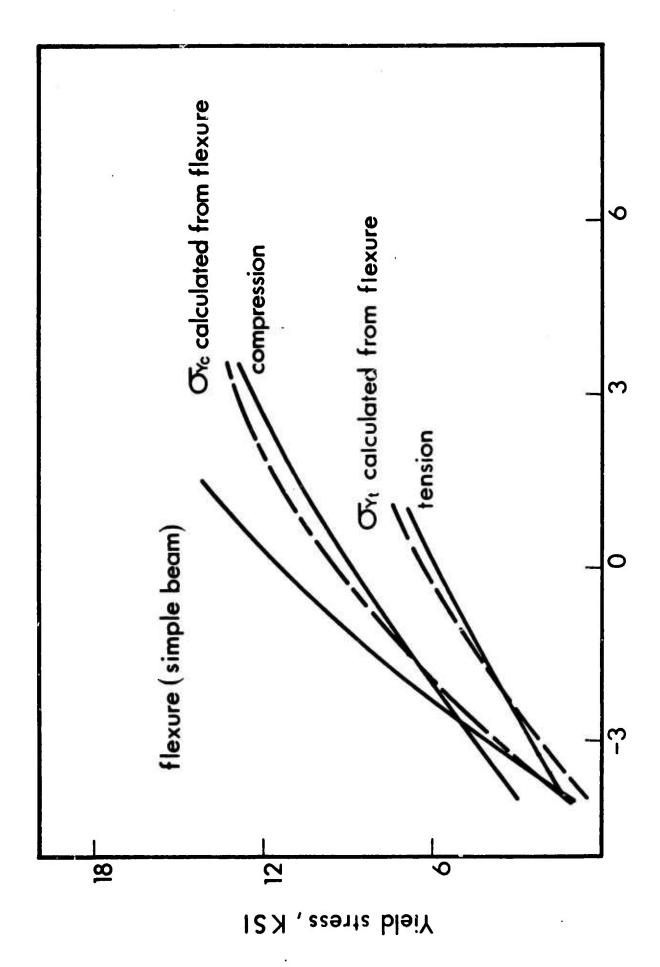


Figure 2i - Yield Stress versus Log Shifted Strain Rate for Particulate Filled,  $T_{\rm ref}$  = 50  $^{\rm o}$ C

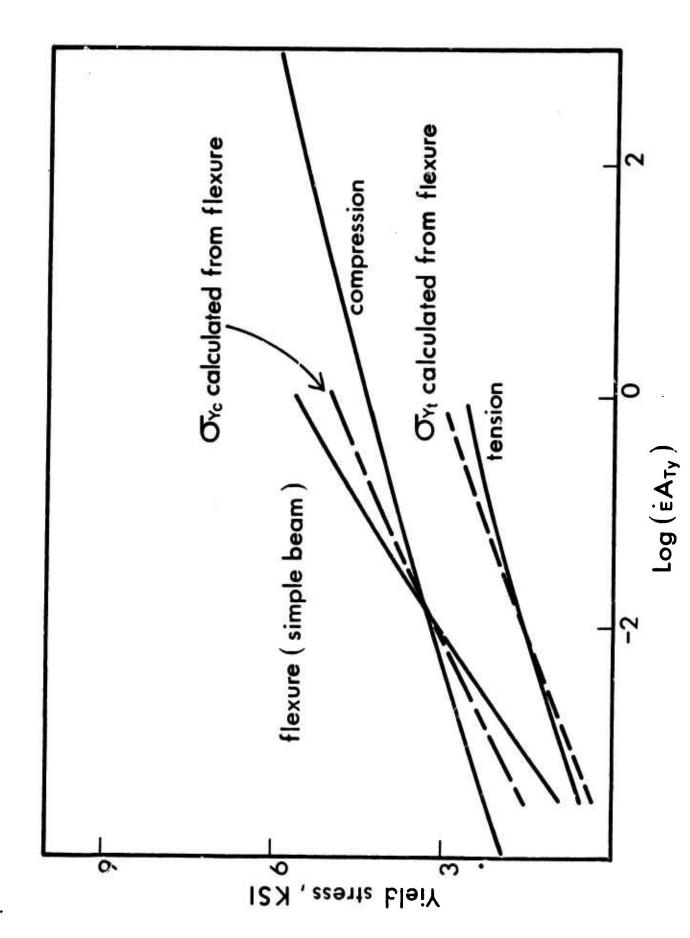


Figure 22 - Yield Stress versus Log Shifted Strain Rate for Foam,  $T_{ref}$  = 50  $^{\circ}$ C

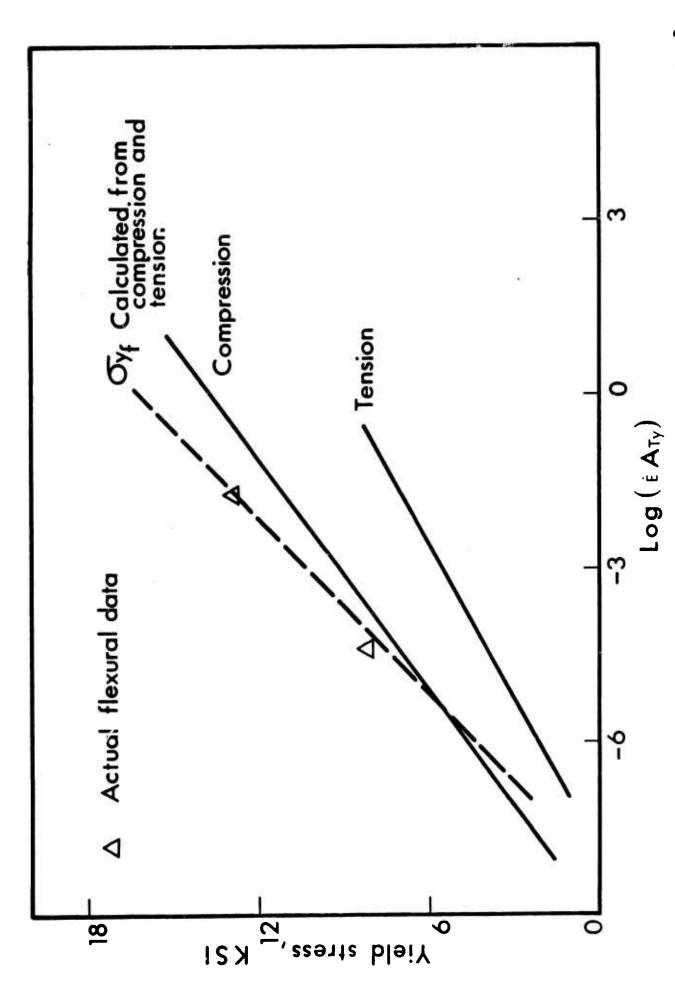


Figure 23 - Yield Stress versus Log Shifted Strain Rate for Continuous Transverse,  $T_{\rm ref}$  = 50  $^{\rm o}$ C

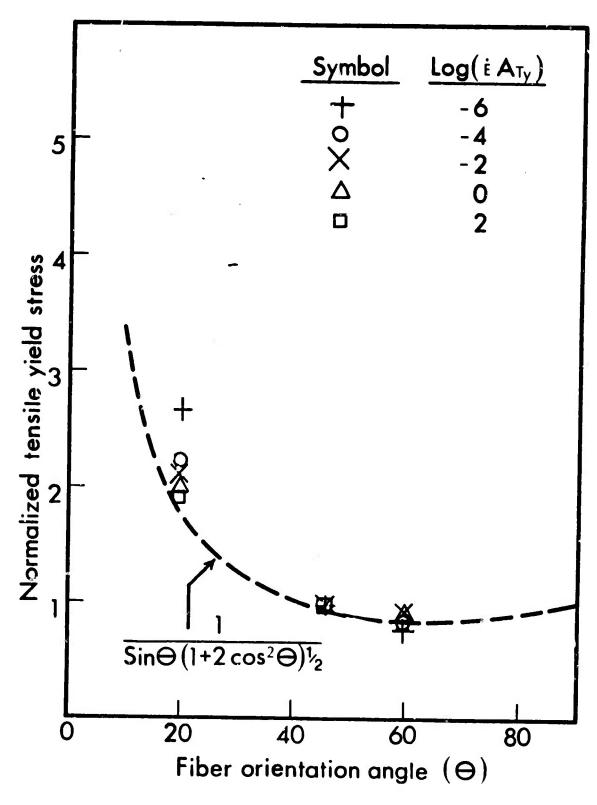


Figure 24 - Normalized Tensile Yield Stress versus Fiber Orientation Angle

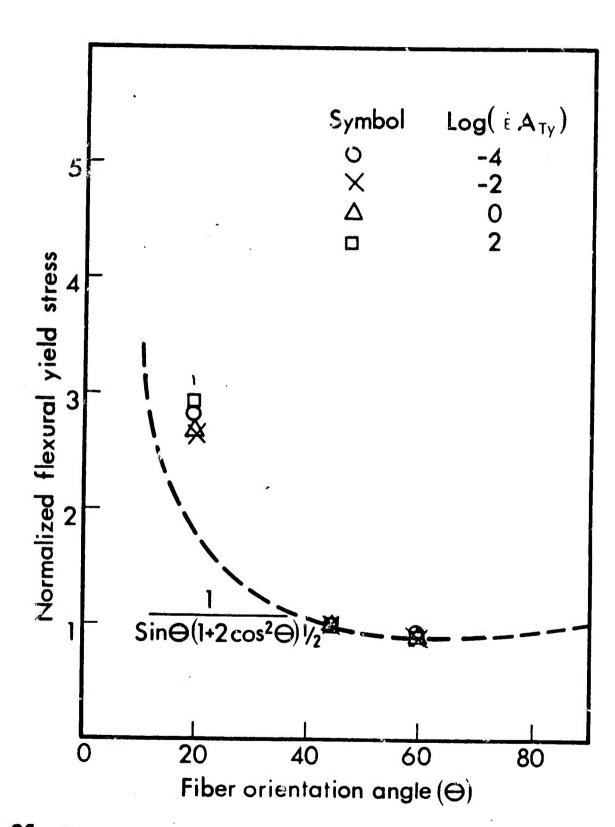


Figure 25 - Normalized Flexural Yield Stress versus Fiber Orientation Angle

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BSTRACT	

A crosslinked epoxy resin consisting of a 60/40 weight ratio of Epon 815 and Versamid 140 and composites of this material with glass beads, unidirectional glass fibers and air (foams) were tested in tension, compression and flexure to determine the effect of time and temperature on the mode of failure, yield stress, and yield strain. Unidirectional continuous fiberfilled samples were tested at different fiber orientation angles with respect to the stress axis. Strain rates ranged from 10<sup>-4</sup> to 10 min<sup>-1</sup> and the temperature from -1 to 107°C.

The material was found to change from a brittle-to-ductile-to-rubbery failure mode with the transition temperatures being a function of strain rate, filler content, filler type and fiber orientation angle, indicating that the transition is perhaps dependent on the state of stress.

In the ductile region, an approximately linear relationship between yieldstress and log strain is evident in all cases. The isotherms of yield stress versus log strain rate were shifted to form a practically linear master plot of yield stress versus log shifted strain rate that can be used to predict the yield stress of the composites at any temperature and strain rate in the ductile region. The time-temperature shift factors were found to be independent of the type, concentration and orientation of filler and the mode of loading. Thus, the composite shift factors seem to be a property of the neutrix and not dependent on the state of stress. The compressive-to-tensile yield stress ratio was practically invariant with shifted strain rate for the unfilled matrix, while fillers and voids raised this ratio and caused it to increase with a decrease in shifted strain rate. The

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yield strain of the composites is less than the unfilled matrix and is a function of fiber orientation and shifted strain rate

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